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IS 10475 (1983): J-acid Urea [PCD 9: Organic Chemicals
Alcohols and Allied Products and Dye Intermediates]

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Indian Standard
SPECIFICATION FOR J-ACID UREA

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INDIAN STANDARDS INSTITUTION
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NEW DELHI 110002

Indian Standard

SPECIFICATION FOR J-ACID UREA

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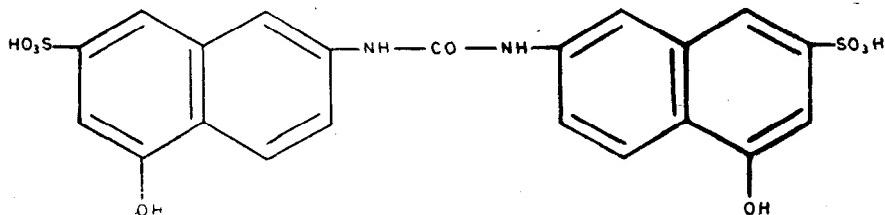
Indian Standard

SPECIFICATION FOR J-ACID UREA

0. F O R E W O R D

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 February 1983, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 J-acid urea ($C_{12}H_{16}O_9N_2S_2$) is an important dye intermediate used for coal tar dyestuffs. The product is available as mono- or di-sodium salt. It is also known as urea of 2-amino-5 naphthol-7 sulphonic acid. It is represented by the following structural formula:



J-ACID UREA
 (Molecular Mass 504·5)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for J-acid urea.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the moist form of light, faintly greyish brown colour.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR J-ACID UREA

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Matter insoluble in sodium hydroxide, percent by mass, <i>Max</i>	0·5	A-1
ii)	Assay, percent by mass, <i>Min</i>	60	A-2
iii)	J-acid content (on 100 percent basis), percent by mass, <i>Max</i>	1·0	A-3

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in fibre drums with polyethylene liners or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Tare, net and gross mass; and
- d) Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Test

4.2.1 Assay content shall be tested on each of the individual samples.

4.2.2 The remaining requirements given in 2 of the specification shall be tested on the composite sample.

4.3 Criteria of Conformity

4.3.1 The lot shall be declared as conforming to the requirements of the standard if all the test results on each of the individual samples and the composite sample meet the relevant specification requirements.

5. METHOD OF TEST

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 **Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(Table 1 and Clause 5.1)

METHOD OF TESTS FOR J-ACID UREA

A-1. DETERMINATION OF MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

A-1.1 Reagent

A-1.1.1 *Sodium Hydroxide Solution* — Dissolve 5 g of caustic soda pellets in water contained in 100-ml measuring flask and make up to the mark.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (second revision).

A-1.2 Procedure — Weigh accurately about 15 g of the thoroughly mixed paste of J-acid urea into a 1-litre breaker. Add about 600 ml of hot water, stir and add sufficient 5 percent sodium hydroxide solution to make it just alkaline to brilliant yellow paper. Boil for a few minutes, filter the hot solution through counter poised filter papers, wash with hot water until free from alkali, dry the papers at 100°C and weigh.

A-1.2.1 Cool the filtrate and washing and transfer to a 1-litre volumetric flask. Dilute to the mark with water and mix well. Call this solution *S* and preserve for assay (*see A-2*).

A-1.3 Calculation

$$\text{Matter insoluble in sodium hydroxide solution, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue; and

M_2 = mass in g of the sample taken for the test.

A-2. ASSAY (BY COUPLING)

A-2.0 Outline of the Method — The material is dissolved in water adding dilute sodium hydroxide solution. A known volume of the solution is titrated against standard benzene diazonium chloride solution in alkaline medium and from the consumption of standard diazonium solution, assay is determined.

A-2.1 Reagents

A-2.1.1 Standard Aniline Diazo Solution — 0·05 N.

A-2.1.2 Sodium Carbonate

A-2.1.3 H-Acid Solution — 1·0 percent.

A-2.1.4 Filter Paper — Whatman No. 1.

A-2.2 Procedure

A-2.2.1 Pipette 50 ml of solution *S* (**A-1.2.1**) into a 500-ml beaker, add 150 ml of ice-cold distilled water, 5 g sodium carbonate and 5 g of sodium chloride. Cool externally in an ice-bath, stir mechanically and titrate at 0 to 3°C with benzene diazonium chloride solution from a cold water-jacketted burette until definite pink colour appears when a drop of titrated solution spotted on filter paper is allowed to diffuse into a drop of faintly alkaline 1 percent H-acid solution after 2 minutes of the last addition.

A-2.3 Calculation

$$\text{Assay, percent by mass} \\ (\text{calculated as J-acid urea}) = \frac{V \times 25.22}{M} \times N$$

where

V = volume in ml of standard benzene diazonium chloride solution,

N = normality of aniline diazo solution (**A-2.1.1**), and

M = mass in g of the sample taken for test.

A-3. DETERMINATION OF J-ACID IN J-ACID UREA BY PAPER CHROMATOGRAPHY

A-3.0 General — A scending paper chromatography is prescribed for determination of J-acid.

A-3.1 Reagents

A-3.1.1 *n-Propanol* — distilled.

A-3.1.2 *Ammonia Solution* — 25 percent *v/v*.

A-3.1.3 *Sulphanilic Acid*.

A-3.1.4 *Sodium Nitrite Solution* — 1 N.

A-3.1.5 *Hydrochloric Acid* — 1 M (see IS : 265-1976*).

A-3.1.6 *Whatman Paper* — 13 mm thickness.

A-3.1.7 *Solvent Mixture* — Developing solvent (*n* propanol and water in 70 : 30 ratio).

A-3.1.8 *J-Acid*.

A-3.1.9 *Sodium Acetate* — Crystals.

A-3.1.10 *Sodium Hydroxide* — 1 N.

A-3.2 Apparatus

A-3.2.1 *Chromatographic Tank* — 27 × 40 cm size.

A-3.2.2 *Sprayer*

A-3.2.3 *Ultra Violet Lamp*

*Specification for hydrochloric acid (second revision).

A-3.3 Preparation of Solutions

A-3.3.1 Standard Sample Solution — Weigh exactly 1·0 g (100 percent) of J-acid urea, dissolve in ammonia solution (A-3.1.2) and make up to 100 ml with distilled water.

A-3.3.2 Standard J-Acid Solution — Weigh exactly 1·0 g (100 percent) of J-acid standard, dissolve in ammonia solution (A-3.1.2) and make up to 1 000 ml with distilled water. From this solution, pipette out 4 ml, 6 ml, and 10 ml in three 100-ml measuring flasks and make up to mark with water. Call this solution *A*, *B* and *C* respectively.

A-3.3.3 Spots

A-3.3.3.1 Sample solution — 0·01 ml is equivalent to 100 micrograms.

A-3.3.3.2 Standard J-acid solution

$$0\cdot01 \text{ ml } (A) = 0\cdot4 \text{ percent}$$

$$0\cdot01 \text{ ml } (B) = 0\cdot6 \text{ percent}$$

$$0\cdot01 \text{ ml } (C) = 1\cdot0 \text{ percent}$$

A-3.4 Preparation of Tank

A-3.4.1 Place 200 ml of developing mixture into the chromatographic tank and replace the lid. Allow to equilibrate for about an hour.

A-3.5 Development of Chromatogram

A-3.5.1 After spotting, suspend the paper from the supporting rods by means of clips so that a lower edge of the paper is just touching the surface of the developing solvent but is not supported on the bottom of the tank. Allow the chromatogram to develop for about 16 hours. After developing and drying at 100°C for 10 minutes develop fluorescence by holding the paper over a beaker containing concentrated ammonium hydroxide solution (25 percent). When the paper is examined under UV lamp the following fluorescence are seen:

- i) Strong blue fluorescence of J-acid urea (main band) at the bottom, and
- ii) Light blue fluorescence of J-acid in the middle.

A-3.5.2 Further assessment shall be made by coupling method as under:

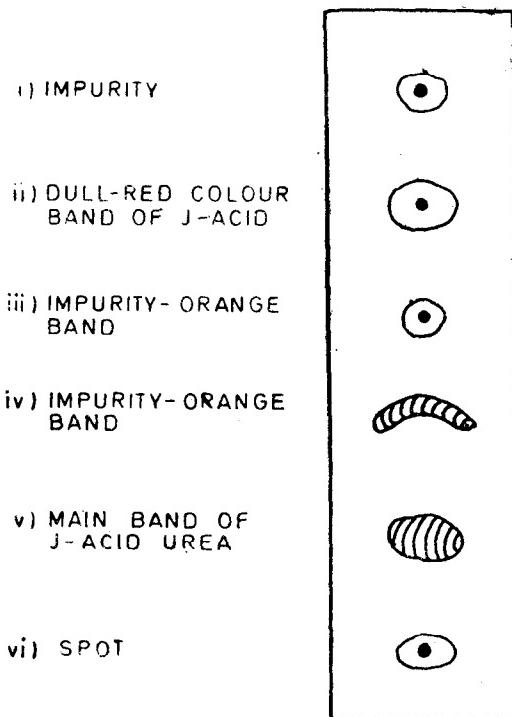
A-3.5.2.1 The chromatogram is coupled by spraying 0·1 N sulphaniilic acid diazo solution. Sulphanilic diazo is prepared as follows:

Weigh approximately 1·7 g of sulphanilic acid, add 15 ml of 1 N sodium hydroxide solution to dissolve and add 30 to 40 ml of

hydrochloric acid (1 M, 30 ml of water and cool to 5°C. To this add previously cooled 10·0 ml of *N* Sodium nitrite solution. Mix well and make up to 100 ml with cold water. The resultant diazo solution should give immediate blue colouration when spotted on starch iodide paper. Add 5·0 g crystalline sodium acetate to the above solution.

A-3.6 Results

A-3.6.1 The following chromatogram develops:



A-3.7 Assessment — Compare the standard spot and evaluate J-acid content.